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IS 8059 (1993): 2-Chloro-5-Aminotoluene-4-Sulphonic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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2-क्लोरो-5-एमिनो टोल्यूइन-4-सल्फोनिक अम्ल, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

2-CHLORO-5-AMINOTOLUENE-4-SULPHONIC ACID, TECHNICAL — SPECIFICATION

(First Revision)

UDC 667·21·547·269'533

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1993

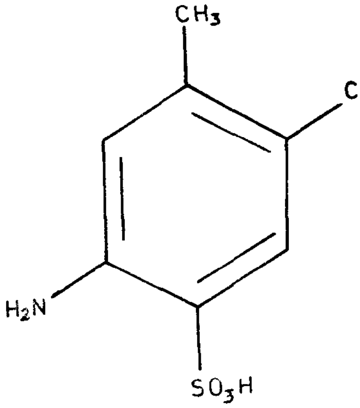
Price Group 3

Dye Intermediates Sectional Committee, PCD 11

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2-Chloro-5-Aminotoluene-4-Sulphonic Acid is an important dye intermediate used in the manufacture of red organic pigments. It is also known as CLT Acid. It has the following structural formula:



2-Chloro-5-Aminotoluene-4-Sulphonic Acid

(Molecular Mass 221·6)

This Indian Standard was first published in 1976. The present revision is being undertaken to include the requirement of 2-chloro-6-aminotoluene-4-sulphonic acid. Besides this a maximum requirement for matter insoluble in sodium carbonate solution and HPLC method for assay determination have been included as an alternate method.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2-CHLORO-5-AMINOTOLUENE-4-SULPHONIC ACID, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for 2-chloro-5-aminotoluene-4-sulphonic acid, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a pinkish white crystalline dry powder. It shall be free from any visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for 2-Chloro-5-Aminotoluene-4-Sulphonic Acid, Technical

(*Clauses* 3.2, 5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Annex)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, (on dry basis), *Min*	97·5	A
ii)	2-Chloro-6-aminotoluene-4-sulphonic acid (leather acid), percent, *Max*	0·5	B
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0·3	C

NOTE — The TLC method (Annex B), for the determination of impurity 2-chloro-6-aminotoluene-4-sulphonic acid (leather acid) is to be performed only when this impurity is available in its pure form.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989), lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed, and shall bear legibly and indelibly the following information:

a) Name of the material,
b) Indication of the source of manufacture,
c) Batch number,
d) Net mass of the material, and
e) Month and year of the manufacture.

4.2.2 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as presented in **3** of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Tests for assay and matter insoluble in sodium carbonate solution shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 The lot shall be declared as conforming to the standard if the tests results as obtained in **5.2.1** and **5.2.2** satisfy the corresponding requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annexes, as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ASSAY

A-1 METHOD A

A-1.1 Outline of the Method

An accurately weighed quantity of the PREPARED sample is dissolved in a an alkaline solution and after diluting with water the total amine is determined by diazotization with standard sodium nitrite solution at 0 to 5°C.

A-1.2 Prepared Sample

Dry the material at 105±1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the material to atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-1.3 Apparatus and Reagents

A-1.3.1 *Glass Beaker* — 2 litre capacity.

A-1.3.2 *Magnetic Stirrer*

Preferably with a teflon rotor.

A-1.3.3 *Sodium Hydroxide Solution*

Approximately 10 percent (m/v).

A-1.3.4 *Hydrochloric Acid*

Approximately 30 percent (m/v).

A-1.3.5 *Standard Sodium Nitrite Solution* — 1 N.

A-1.3.6 *Starch-Potassium Iodide*

Paste or starch-iodide indicator papers.

A-1.4 Procedure

Weigh accurately about 5·000 g of the prepared sample (*see* **A-1.1**) and transfer into a 2-litre beaker containing about 300-ml of water. Dissolve the substance by the addition of 15 ml of sodium hydroxide solution, to a light yellowish brown clear solution. Heat the mixture, if necessary to obtain a clear solution. Allow the solution to cool to room temperature and acidify with 20 ml of hydrochloric acid. The solution remains nearly clear. Add ice to it to cool to 0°C and titrate against standard sodium nitrite solution until a spot on the starch-iodide paper gives a faint but distinct blue colouration. This blue colouration should persist for 5 minutes without further addition of the nitrite solution.

A-1.4.1 *Calculation*

$$\text{Assay, percent by mass} = \frac{V \times N \times 22{\cdot}16}{M}$$

where

V = Volume in ml of the standard sodium nitrite solution consumed for complete diazotization,

N = Normality of the standard sodium nitrite solution, and

M = Mass in g of the material taken for test.

A-2 METHOD B (Alternate Method)

A-2.1 General

CLT acid and its impurities are estimated by high pressure liquid chromatographic method.

A-2.2 Reagents

A-2.2.1 *Acetonitrile* — HPLC grade.

A-2.2.2 *Water* — Double distilled, HPCL grade.

A-2.2.3 *Sodium Dihydrogen Phosphate*, AR grade.

A-2.2.4 *2Chloro-5-Aminotoluene-4-Sulphonic Acid, Technical* — Pure (CLT acid).

A-2.2.5 *2-Chloro-6-Aminotoluene-4-Sulphonic Acid* — Pure (Leather acid).

A-2.3 Apparatus

A-2.3.1 *High Pressure Liquid Chromatograph*

Any suitable chromatograph with a reverse phase column. A typical parameter of water's HPLC is given below:

i)	Column	: Stainless steel, 125 mm × 4·9 mm Nucleosil C_{18}, 5µ particle size
ii)	Temperature	: 25°C
iii)	Mobile phase	: Aqueous Sodium dihydrogen phosphate (0·05 M) + Acetonitrile (92 : 8)
iv)	Flow rate	: 1·0 ml/min
v)	Injection volume	: 5 µl
vi)	Chart speed	: 5 mm/min
vii)	Attenuator	: 1·0
viii)	Wave length (Detector)	: 254 nm

A-2.4 Preparation of Solutions

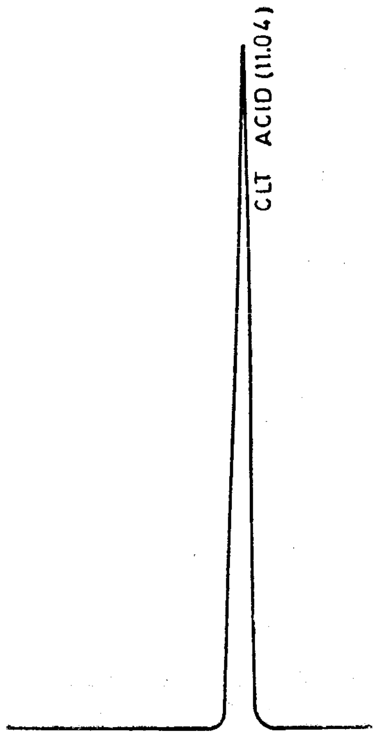
A-2.4.1 *Preparation of Sample Solution*

Weigh accurately 0·03 g of prepared sample (*see* **A-1.2**) and transfer to a 100 ml volumetric flask and dissolve the sample in the mobile phase (aqueous solution of sodium dihydrogen phosphate (0·05 M) and acetonitrile (92 : 8) and prepare the solution by making up to the mark. Stopper and shake well.

A-2.4.2 *Preparation of Standard Solution*

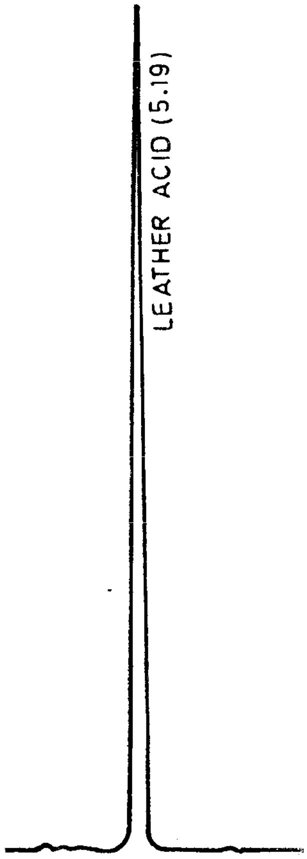
A-2.4.2.1 *2-Chloro-5-aminotoluene-4-sulphonic acid, technical* — Pure.

Weigh accurately 0·03 g of pure 2-chloro-5-aminotoluene-4-sulphonic acid and transfer to 100 ml volumetric flask and prepare the solution by making up to the mark using the sodium dihydrogen phosphate (0 05 M) buffer solution and acetonitrile (92 : 8). Stopper and shake well.



PEAK	AREA%	RT	AREA	BC
1	0·008	1·3	3224	01
2	0·013	1·46	5079	01
3	0·009	1·7	3440	01
4	0·019	3·28	7122	01
5	0·01	5·67	3855	01
6	99·818	11·04	38385206	02
7	0·123	12·9	47362	03
TOTAL	100		38455288	

FIG. 1 TYPICAL CHROMATOGRAM FOR CLT ACID



PEAK	AREA%	RT	AREA	BC
1	0·105	1·29	25039	02
2	0·025	1·71	5903	02
3	0·12	1·86	28796	02
4	0·021	2·24	5077	02
5	0·006	2.45	1473	02
6	0·01	2·51	2508	02
7	0·011	2·64	2651	03
8	0·015	3·2	3692	01
9	99·677	5·19	23825046	02
10	0·008	6·64	1983	03
TOTAL	100		23902168	

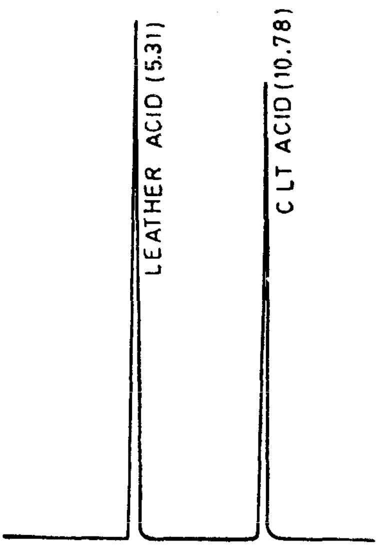
FIG. 2 TYPICAL CHROMATOGRAM FOR LEATHER ACID

A-2.4.2.2 *2-Chloro-6-aminotoluene-4-sulphonic acid* — Pure.

Weigh accurately 0·03 g of pure-2-Chloro-6-Aminotoluene-4-Sulphonic acid and transfer to 100 ml volumetric flask. Prepare the solution by making up to the mark using the sodium dihydrogn phosphate (0·05 M) buffer solution and acetonitrile (92 : 8). Stopper and shake well.

A-2.5 Procedure

When steady baseline is obtained, inject 5·0 μl of each standard solution and also inject 5·0μl of sample solution. Compute the area of each peak and calculate. Typical chromatogram for CLT acid, leather acid and (CLT acid + Leather acid) are given in Fig. 1, 2 and 3 respectively.



PEAK	AREA%	RT	AREA	BC
1	0·053	4·65	4201	01
2	53·628	5·31	4242389	01
3	0·015	10·11	1168	01
4	46·305	10·78	3663080	01
TOTAL	100·		7910838	

FIG. 3 TYPICAL CHROMATOGRAM FOR CLT ACID + LEATHER ACID

A-2.6 Calculation

$$\text{CLT acid, percent by mass (on dry basis)} = \frac{\text{Concentration of standard} \times \text{Area of the peak in the sample} \times \text{Purity of standard}}{\text{Concentration of sample} \times \text{Area of peak of standard}}$$

ANNEX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF 2-CHLORO-5-AMINOTOLUENE-4-SULPHONIC ACID (LEATHER ACID)

B-1 GENERAL

Estimation of 2-chloro-6-aminotoluene-4-sulphonic acid (leather acid) is done by thin layer chromatography. However, this estimation should be done only when a pure form of this impurity is available.

B-2 APPARATUS

B-2.1 Thin Layer Chromatographic Plate

Glass plate of size 20 cm × 20 cm coated uniformly with silica gel and freshly activated for about 1 hour at 90°C.

B-2.2 Micropipette

10 µl capacity.

B-2.3 Developing Chamber

B-2.4 Chromatographic Sprayer

B-3 REAGENTS

B-3.1 2-Chloro-5-Aminotoluene-4-Sulphonic Acid — Pure.

B-3.2 2-Chloro-6-Aminotoluene-4-Sulphonic Acid — Pure.

B-3.3 Dimethyl Sulphoxide

Analytical reagent grade.

B-3.4 Developing Solvent

Ethyl Acetate : Ethanol : Aqueous Ammonia: Water (94 : 4 : 0·2 : 1·8).

B-3.5 Spray Solution

A methanolic solution of American base (N-1-Naphthylethylene diamine dihydrochloride) 0·5 percent (*m*/*v*).

B-4 PROCEDURE

B-4.1 Weigh accurately 1·0 g of the prepared sample (**A-1.2**) and dissolve it in 50 ml of dimethyl sulphoxide and transfer to a 100 ml volumetric flask and dilute to 100 ml with dimethyl sulphoxide.

B-4.2 Weigh accurately 1·0 g of the pure 2-chloro-5-aminotoluene-4-sulphonic acid and dissolve in dimethyl sulphoxide as in **B-4.1** and transfer to a 100 ml volumetric flask. Add 5·0 ml of 0·1 percent solution of 2-chloro-6-aminotoluene-4-sulphonic acid in dimethyl sulphoxide to the volumetric flask and dilute to 100 ml with dimethyl sulphoxide.

B-4.3 Spot 10 µl each of the sample solution (**B-4.1**) and the reference solution (**B-4.2**) on the thin layer plate. Dry well with the help of an air drier, Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in the ascending manner to a distance past the starting line. Remove the plate from the chamber and dry off the solvent in an air drier. Hold the plate for 20 seconds in nitrous acid fumes (formed by adding about 5 g of sodium nitrite to 50 ml of 5 N hydrochloric acid in a 5 litre beaker). Allow the plate to stand for 1 minute to remove excess nitrous fumes. Spray with the spray solution and after a few minutes examine visually the intensity of colour of each zone developed with the sample solution and compare it with that developed with a reference solution. (Rf value of 2-chloro-5-aminotoluene-4-sulphonic acid is about 0·55, Rf value of 2-chloro-6-aminotoluene-4-sulphonic acid is about 0·46).

ANNEX C

[*Table* 1, *Item* (iii)]

MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

C-1 REAGENT

Sodium Carbonate — 2 percent (*m*/*v*).

C-2 PROCEDURE

C-2.1 Weigh accurately 5·00 g of 2-Chloro-5-aminotoluene-4-Sulphonic acid and transfer to a 500 ml beaker. Add 100 ml of 2 percent sodium carbonate solution. Heat the beaker to about 75 to 80°C in a steam bath till most of the material is dissolved. Observe the oplaescence. If the insoluble matter is more than a trace, filter through a previously washed dried and weighed sintered crucible of porosity G-4. Wash thoroughly the matter on the sintered crucible with water till alkali free, dry and weigh. Repeat the operation of drying and weighing until constant weight is obtained. Calculate the percentage of alkali insoluble matter according to the formula.

C-3 CALCULATION

$$\text{Matter insoluble in sodium carbonate solution, percent by mass} = \frac{m \times 100}{M}$$

where

m = mass of residue, and

M = mass of material taken for test.

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